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CHEMICAL AND PHYSICAL CHARACTERIZATION OF NITROCELLULOSE FINES

SPECIAL REPORT

by

Danny O. Helton

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The findings of this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

For

Dr. David H. Rosenblatt
Environmental Protection Research Division
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Techniques were developed to optically differentiate cellulose or acetylated cellulose esters from nitrocellulose. The sample could be readily fractionated by passing the slurry through sieves and membrane filters of different "Particle Size" ranges. Based on "Particle Size" distribution and nitro content over 99% of the total sample was military grade nitrocellulose with an average nitrogen content of 12.9%.

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When assayed by differential thermal analysis (DTA), all of the "Particle Size" ranges gave a characteristic exotherm at about 195 to 210°C. This exotherm is a characteristic of the nitrate ester functionability. The < 0.2 µ "Particle Size" sample had a caloric value on a weight basis of < 10% of that of the > 88 µ sample, thus indicating the presence of low nitro content nitrocellulose.

When the particles were assayed for nitration by direct nitro content assay, the "Particle Size" ranges above 0.8 µ were observed to be 82 to 95% nitrated, while the particle ranges of < 0.8 µ were about 30% or less nitrated. Percent nitration by elemental analysis indicated values which were in reasonable agreement with the nitro content assays for "Particle Sizes" > 0.8 µ. Below 0.8 µ there was serious disagreement between the percent nitration by nitro content and elemental analysis data. The elemental analysis data for the < 0.8 µ samples suggested 60% nitration versus 30% by nitro content. Since the elemental analysis determination is the sum of the nitrogen content from sources other than nitrate ester, such as inorganic nitrate and nitrite, the chemical reduction assay is considered more reliable. When combined, the DTA data and the nitro content assays indicated the 0.8 to 0.2 µ "Particle Size" sample was low nitro content nitrocellulose.

Molecular weight distribution data indicate "Particle Size" ranges above 44 µ have typical military grade nitrocellulose M_n, M_w, and M_w/M_n values while "Particle Size" ranges of less than 44 µ show abnormally low M_n values and high M_w/M_n ratios.

PREFACE

This report was prepared at Midwest Research Institute, 425 Volker Boulevard, Kansas City, Missouri, 64110, under Department of the Army, Contract No. DAMD-17-74-C-4073, MRI ~~Project No. 3900~~-B.

The proposed work was outlined in our Supplement to Renewal Proposal No. B-1657 (Section III, F.4) of March 10, 1975. The work was supported by the Environmental Protection Research Division, U.S. Army Medical Research and Development Command, Department of the Army. Captain John P. Glennon of the Environmental Protection Research Department, U.S. Army Medical Bioengineering Research and Development Laboratory, Fort Detrick, Maryland, 21701, was the contract monitor for this project.

The work was conducted in the Biological Sciences Division, under the direction of Dr. William B. House, and the Physical Sciences Division, under the direction of Dr. Harold M. Hubbard, between April and November 1975. The experimental work was supervised by Dr. Danny O. Helton, Senior Chemist, with the technical assistance of Bernadette Chipko, Assistant Chemist; Jean Cahoy, Assistant Chemist; and John Rollheiser, Assistant Chemist.

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I. INTRODUCTION

This work was performed in support of the overall nitrocellulose (NC) toxicology effort being performed at Midwest Research Institute. While the literature provides ample data on the chemical and physical properties of military grade nitrocellulose, very little is reported on the by-products. Earlier work at MRI indicated the nitrocellulose fines (< 10 microns) may be more toxic than the larger particles.

The Ammunition Procurement and Supply Agency (APSA) proposed wastewater effluent standard requires a suspended solids concentration of less than 25 ppm. The final engineering report on Project PE-221 (Radford Army Ammunition Plant; Hercules, Inc.) indicates the suspended solids concentration variation is 80 to 900 ppm, and that from B line alone, 2 tons of solids per day are entering the effluent from the line. The suspended solids analyzed more than 99% nitrocellulose (NC). About 90% of the solids were smaller than 10 microns (μ).

Numerous methods have been studied for the separation of nitrocellulose. These methods include: (a) settling pits; (b) continuous centrifugal separation; (c) liquid cyclone; (d) air flotation; (e) filtration; and (f) flocculation. The final engineering report on Project PE-221 indicates that these methods are nonfunctional or would cost several hundred thousand dollars per year to operate.

The following pages discuss the program objective, physical and chemical characterization procedures, conclusions, and recommendations.

II. OBJECTIVE

The objective of this project is to obtain chemical and physical characterization data on the nitrocellulose waste generated at Radford Army Ammunition Plant. Physical characterization methods include particle size distribution, selective staining techniques, molecular weight distribution, and differential thermal analysis. Chemical characterization includes sulfate content, nitro content, and elemental analysis.

III. PHYSICAL CHARACTERIZATION

The waste nitrocellulose was fractionated by wet sieving and characterized by weight distribution according to "particle size," particle length, optical methods using stains or dispersion techniques, molecular weight distribution, and differential thermal analysis.

A. Sampling Procedure

The samples were obtained from Line B (cotton linters) Poacher Pits at Radford Army Ammunition plant. The material was shipped to MRI in 55-gal. barrels. Samples were removed for testing after stirring the barrel contents. Some obvious "impurities" such as leaves, twigs, etc., were removed prior to further work.

Data were obtained using two Lots of nitrocellulose. The lots differed in sampling procedure only in that they were taken from Line B a few months apart.

B. Weight Distribution by "Particle Size"

The sample was fractionated according to the ability of the particles to pass through sieves and filters. The sample was wet sieved using 88-μ and 44-μ sieves. The particles passing the 44-μ sieve were fractionated using Gelman Instrument Company Metricel™ membrane filters of 5, 0.8, and 0.2 μ. References to "Particle Size" indicate the ability of particles to pass through various sieves and filters. The results using Lot 1 were:

TABLE I

WEIGHT DISTRIBUTION AS A FUNCTION OF "PARTICLE SIZE"

"Particle Size" (μ)	Weight Distribution (%)
> 88	66
< 88 and > 44	23
< 44 and > 5	11
< 5 and > 0.8	0.1
< 0.8 and > 0.2	< 0.1
< 0.2	0.1

The weight distribution by "Particle Size" is expected to change as a function of sampling site, water flow rate, whether the starting material is wood pulp or cotton, etc.

C. Particle Length

The lengths of the fibers in the various "Particle Size" ranges were determined. The data were obtained by photographing suspensions of the particles followed by direct measurements using a calibrated flexible ruler. The results, using Lot 1, are shown in Figures 1, 2, and 3.

For the "particle size" > 88 μ, 88% of the particles were 0.2 to 0.9 mm long; the average length was 0.63 ± 0.30 mm, and the most common length was 0.4 to 0.5 mm. For the "particle size" < 88 and > 44 μ, 90% of the particles were 0.05 to 0.45 mm long; the average length was 0.27 ± 0.19 mm,

and the most common length was 0.15 to 0.20 mm. For the "particle size" < 44 and > 5 μ, 90% of the particles were 0.05 to 0.13 mm long; the average length was 0.006 ± 0.044 mm, and the most common length was 0.02 to 0.3 mm. "Particle size" ranges below 5 μ were not studied.

For 10 fibers randomly chosen from the "particle size" ranges > 88 μ, < 88 to > 44 μ, and < 44 to > 5 μ the average diameter was 31 ± 4 μ.

D. Optical Methods

Cellulose can be optically differentiated from nitrocellulose by simple staining or dispersion techniques. The techniques are discussed below.

1. Dispersion staining techniques

a. Sample preparation

A few drops of fiber-in-water slurry was placed on a microscope slide and was covered with a cover slip. The slide was placed on a hot plate (80-90°C) until all water had evaporated. A drop of Cargille liquid of refractive index 1.520 was introduced under the cover slip.

b. Examination

The sample was viewed through a dispersion staining objective.

c. Results

Cellulose appeared yellow and nitrocellulose appeared bright blue.

2. Sample staining techniques

a. Dyes

Pontamine Fast Blue RRL dye (0.1 gm) and Celliton Fast Yellow RRA (0.1 gm) were dissolved in 50 ml of water.

(1) Sample preparation

The sample containing cellulose and nitrocellulose was allowed to stand 15-18 hr in the dye. The dye was then decanted and the fibers were washed in several volumes of water until excess dye had washed out. The final wash was not decanted. A few drops of the final wash solution were transferred to a microscope slide and dispersed by dropping a cover slip in place.

(2) Examination

The sample was viewed using transmitted illumination.

(3) Results and discussion

The nitrocellulose fibers are stained pale yellow, and the cellulose fibers are stained blue. Pontamine blue is specific for cellulose while celliton yellow is general for esters of cellulose such as nitrocellulose and cellulose acetate. The attached photographs (Figures 4 through 7) and comments in Table II illustrate the use of this dye.

TABLE II

COMMENTS ON NITROCELLULOSE DYES

Figure	Comment
4	Mixture of untreated cellulose and nitrocellulose
5	Nitrocellulose treated with dye
6	Cellulose treated with dye solution
7	Nitrocellulose and cellulose fibers treated with dye

b. Nitrite specific reagent

When sodium hydroxide reacts with nitrocellulose, both nitrate and nitrite are produced. The liberated nitrite can be detected by reaction with sulfanilic acid and N-(1-naphthyl)ethylenediamine to form a rose-colored product. This reaction was used to stain nitrocellulose as discussed below.

(1) Sample preparation

A few drops of the sample in water were placed on a microscope slide and allowed to evaporate. The slide was sprayed with a fine mist of 1 N NaOH, allowed to dry, and then sprayed with a 1 N HCl solution containing 1% sulfanilic acid and 1% N-(1-naphthyl)ethylenediamine. After allowing the solvent to evaporate, Cargille liquid of refractive index 1.700 was added.

(2) Results

In most cases the nitrocellulose fibers appeared in bright magenta color, while the cellulose remained semitransparent.

When mixtures of cellulose and nitrocellulose were examined there were some fibers that exhibited only slight coloration. In these later cases, it could not be determined if the color resulted from the bleeding of stained fibers to cellulose or if the color was a positive test for nitrocellulose. The mobility of the stain was apparent from the presence of a magenta halo surrounding many of the stained fibers. The attached photographs (Figures 8 through 11) and the comments in Table III illustrate this dye.

TABLE III

COMMENTS ON NITRITE SPECIFIC STAIN

Figure	Comment
8	Cellulose treated with stain
9, 10	Nitrocellulose treated with stain
11	Mixture of cellulose and nitrocellulose with stain

E. Molecular Weight Distribution

The molecular weight distribution of the fractions was determined by gel permeation chromatography (GPC) using methods suggested by Nauflett and French,[1/] and Chang, et al.[2/] The operating procedures and results are given below.

1. Standard conditions for operation of Waters gel permeation chromatograph

Solvent: Tetrahydrofuran

Flow Rate: 1.00 ml/min

Sample Columns (4 ft x 3/8 in. O.D., μStryragel): 10^6 Å, 10^5 Å, 10^4 Å, 10^3 Å, and 10^2 Å.

Injection Procedure: 100 μl of an ~ 0.5% by weight solution using a syringe.

Instrument: Waters Associates 201 equipped with refractive index and ultraviolet (254 nm) detectors.

2. Calibration of columns

The columns were calibrated using Waters Associates' polystyrene standards. The GPC curves were analyzed by the method of Chang, et al.[2/] The results are shown in Table IV.

TABLE IV

GPC RESULTS ON POLYSTYRENE STANDARDS

Waters' Cat. No.	Molecular Weight M_w	Molecular Weight M_n	Molecular Weight Peak M	Elution Volume, ml Counts Peak M
61970	2,610,000	1,990,000	2,300,000	31.5
25166	411,000	392,000	402,000	35.0
41984	111,000	111,000	111,000	38.5
25170	36,000	33,000	34,500	41.0
25171	10,300	9,700	10,000	44.0
41985	2,020	2,020	2,020	48.0

The polystyrene curve satisfied the equation:

$$\log M_n = \log a + mc = 12.245 - 0.187\ C$$

where M_n = molecular weight by number average

C = elution volume in ml

The preliminary nitrocellulose curve was obtained using the data of Chang, French et al.[2/] and Carignan and Turngen.[3/] The M_n values were calculated and the final curves drawn. The nitrocellulose calibration curve satisfied the equation:

$$\log M_n = \log a + mc = 12.351 - 0.198\ C$$

where M_n = molecular weight by number average

C = elution volume in ml

3. Results

The results are shown in Table V.

TABLE V

MOLECULAR WEIGHT AS A FUNCTION OF "PARTICLE SIZE"

"Particle Size"(μ)	M_n	M_w	M_w/M_n (heterogeneity)
> 88	37,200	165,000	4.4
< 88 and > 44	54,000	158,000	2.9
< 44 and > 5	7,770	167,000	21.5
< 5 and > 0.8	8,690	110,900	12.8

The cumulative distribution plots for this data are shown in Figures 12, 13, 14, and 15. The precision for the M_n and M_w values is estimated to be ± 5%.

By comparison, Nauflett and French[1/] studied 11 military grade nitrocellulose samples where the nitrogen content varied from 11.86 to 13.13, the M_n varied from 22,100 to 76,500, the M_w varied from 57,900 to 478,900, and the M_w/M_n ratio varied from 2.62 to 8.40.

The 0.8 to 0.2 μ and < 0.2 μ samples gave the same peaks as a tetrahydrofuran extracted cotton linter sample, thereby suggesting the absence of nitrocellulose. However, cellulose with 30% or less nitration is not expected to be soluble in tetrahydrofuran, and the 0.8 to 0.2 μ and < 0.2 μ samples were not appreciably soluble in tetrahydrofuran.

The > 88 μ "Particle Size" sample showed refractive index and UV peaks at 51.5 and 56 ml elution volume. The 88 to 44 μ and 44 to 5 μ sample showed a refractive index and UV peak at 56 ml elution volume. The compounds with elution volumes of 51.5 and 56 ml were probably produced during the manufacture of nitrocellulose and trapped within the fibers. Peaks of 50 ml or more elution volume were not within the linearity range of the calibration curve, but probably represent molecular weights of ≤ 1,000.

A sample of cotton linters used for the preparation of nitrocellulose was extracted with tetrahydrofuran and the extract by GPC. No peak with an elution volume of < 50 ml was observed.

F. Differential Thermal Analysis

The fractionated samples were studied using a Du Pont 900 Differential Thermal Analysis instrument. Sample sizes of 1 to 2 mg were used. The results were shown in Table VI.

TABLE VI

DTA ANALYSIS AS A FUNCTION OF "PARTICLE SIZE"

"Particle Size" (μ)	Lot 2 Exotherm	Lot 2 Endotherm
> 88	193-209°	---
88 to 44	195-209°	---
44 to 5	197-207°	---
5 to 0.8	198-208°	---
0.8 to 0.2	193-210°	---
< 0.2	196-211° [a]	366-400°

a/ The exotherm at 196-211° had less than 10% of the caloric value of that for the > 88 μ sample.

All samples except the < 0.2 μ sample were off-white in color. The < 0.2 μ sample on heating changed from light brown to dark brown, but otherwise appeared only slightly changed. The other samples on heating become a black charred mass.

These data indicate that all samples above 0.2 μ contain an appreciable concentration of the nitrate ester functionality.

IV. CHEMICAL CHARACTERIZATION

The fractionated material was studied according to the ability of the particles to pass through various sieves and filters as indicated in Section II.B. References to "Particle Size" indicate the ability of particles to pass through various sieves and filters.

The samples are characterized by sulfate content, nitro content, and elemental analysis.

A. Sulfate Content

The samples were oxidized with nitric acid-perchloric acid-magnesium nitrate reagent according to the method of Norwitz.[4] The ionic sulfate concentration was determined by the automated methylthymol blue method described by the Environmental Protection Agency.[5] Using Lot 1 the results were:

"Particle Size" (μ)	Percent Sulfate/Gram Line B Sample
> 88	0.11
< 88 and > 44	0.24
< 44	0.29

Although the military specifications for nitrocellulose do not specify a maximum allowable concentration of sulfate,[6] these concentrations are not expected to cause abnormal instability.[7]

B. Nitro Content

The reducible NO_2 content of the fractions was determined by the chemical reduction method of Selig.[8] The results were:

"Particle Size" (μ)	Sample from Lot 1	
	% N as NO_2[a]	% Nitration[a]
> 88	12.87	91 ± 2
< 88 and > 44	13.43	95 ± 2
< 44 and > 5	11.59	82 ± 2
< 5 and > 0.8	11.59	82 ± 2
< 0.8 and > 0.2	4.10	29 ± 1
< 0.2	3.39	24 ± 1

a/ Average of two determinations.

The calculation for percent nitration assumes nitrocellulose to be the only source of reducible NO_2 groups. This assumption is probably a reasonable one for those particles which can be filtered and washed, i.e., all except the < 0.2 μ sample. The < 0.2 μ sample can contain inorganic nitrate and nitrite.

C. Elemental Analysis

The fractions were analyzed for elemental content.[9] The results were:

"Particle Size" (μ)	Lot 2 % C	% H	% N	% Nitration by % N
> 88	26.46	2.16	12.16	86
< 88 and > 44	25.57	2.45	12.56	89
< 44 and > 5	27.28	3.09	10.31	73
< 5 and > 0.8	26.86	2.61	11.70	83
< 0.8 and > 0.2	30.89	3.86	8.86	63
< 0.2	5.01	0.70	7.58	54

The percent nitration as calculated from percent nitrogen content for > 88 μ, < 88 and > 44 μ, < 44 and > 5 μ, is 6 to 9% lower than the percent nitration by direct nitro content assay (see Section B above). This fluctuation may well be due to variations from Lot 1 to Lot 2. However, the percent nitration by elemental analysis for the < 5 μ samples is widely variant to that obtained by direct nitro content assay. The differential thermal analysis data also suggests the < 0.2 μ sample is not ~ 50% nitrated. Thus, for the < 5 μ samples, the percent nitrogen by elemental analysis is a poor determination of the percent nitration. The low C, H, and N content of the < 0.2 μ sample suggests this sample has a high inorganic component(s) content.

V. DISCUSSION AND CONCLUSIONS

Techniques were developed to optically differentiate cellulose or acetylated cellulose esters from nitrocellulose. The sample could be readily fractionated by passing the slurry through sieves and membrane filters of different "Particle Size" ranges. Based on "Particle Size" distribution and nitro content over 99% of the total sample was military grade nitrocellulose with an average nitrogen content of 12.9%.

When assayed by differential thermal analysis (DTA), all of the "Particle Size" ranges gave a characteristic exotherm at about 195 to 210°C. This exotherm is a characteristic of the nitrate ester functionability. The < 0.2 μ "Particle Size" sample had a caloric value on a weight basis of < 10% of that of the > 88 μ sample, thus indicating the presence of low nitro content nitrocellulose.

When the particles were assayed for nitration by direct nitro content assay, the "Particle Size" ranges above 0.8 μ were observed to be 82 to 95% nitrated, while the particle ranges of < 0.8 μ were about 30% or less nitrated. Percent nitration by elemental analysis indicated values which were in resonable agreement with the nitro content assays for "Particle Sizes" > 0.8 μ. Below 0.8 μ there was serious disagreement between the percent nitration by nitro content and elemental

analysis data. The elemental analysis data for the < 0.8 μ samples suggested 60% nitration versus 30% by nitro content. Since the elemental analysis determination is the sum of the nitrogen content from sources other than nitrate ester, such as inorganic nitrate and nitrite, the chemical reduction assay is considered more reliable. When combined, the DTA data and the nitro content assays indicated the 0.8 to 0.2 μ "Particle Size" sample was low nitro content nitrocellulose.

Molecular weight distribution data indicate "Particle Size" ranges above 44 μ have typical military grade nitrocellulose M_n, M_w, and M_w/M_n values while "Particle Size" ranges of less than 44 μ show abnormally low M_n values and high M_w/M_n ratios.

VI. RECOMMENDATIONS

Current toxicity data generated at MRI indicate nitrocellulose has low toxicity in mammalian systems. Thus, further characterization is not warranted at this time. Should long-term feeding of nitrocellulose to rodents produce toxicity, carcinogenicity, etc., further work is justified. Future characterization work might well be influenced by the type of animal ailment but would probably concentrate on analysis of the NC fines. Moderate amounts of NC fines (> 1 g) should be obtained by on-site sampling, then characterized by techniques such as IR, UV, NMR, carboxyl content, etc. Methods for removing these fines, such as electrolysis or reverse osmosis, should also be studied.

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9. Assayed by Galbraith Laboratories, Inc., Knoxville, Tennessee.

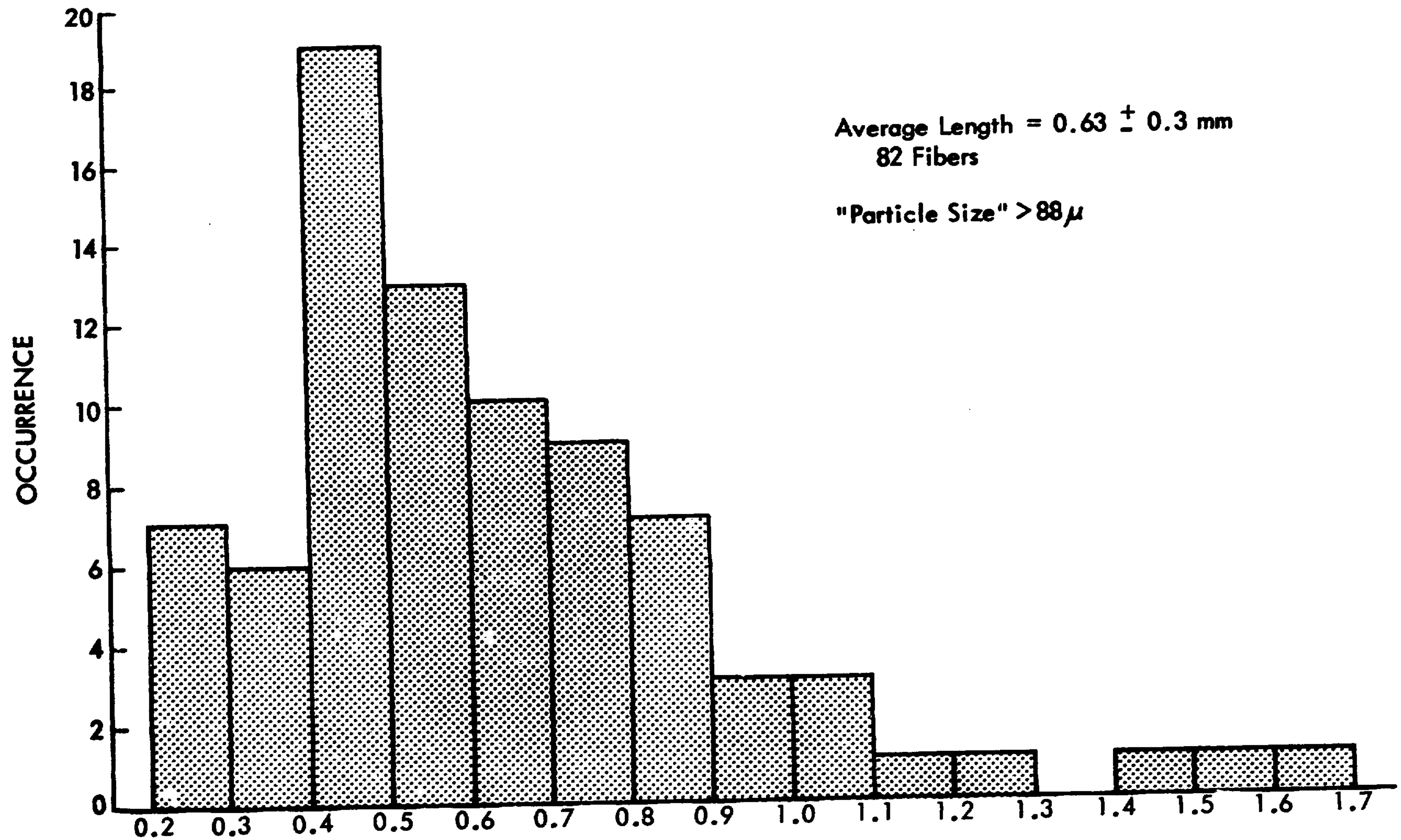

Figure 1 - PARTICLE LENGTH ,mm

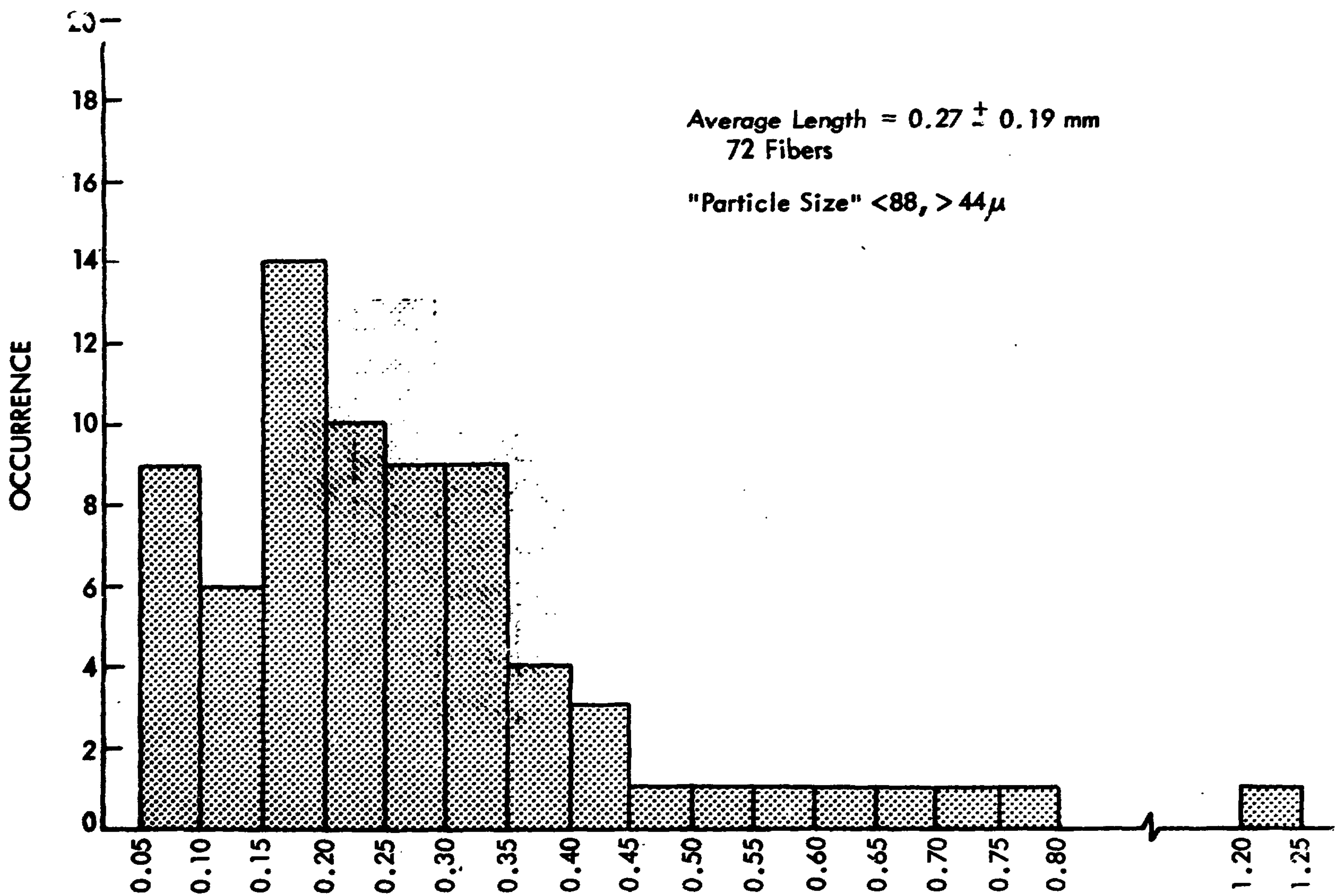

Figure 2 - PARTICLE LENGTH ,mm

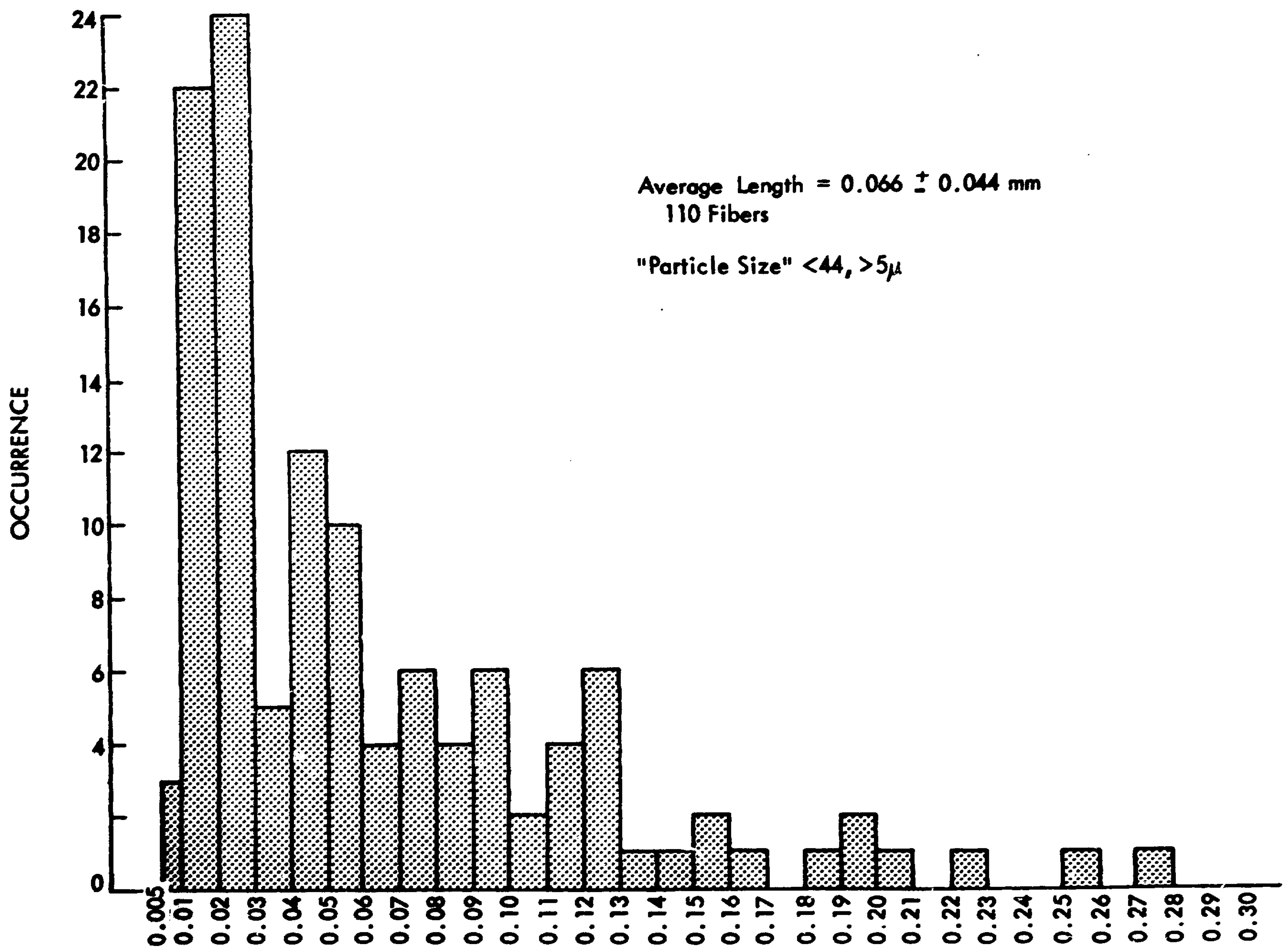

Figure 3 - PARTICLE LENGTH ,mm

Figure 4 - Mixture of Untreated Cellulose and Nitrocellulose

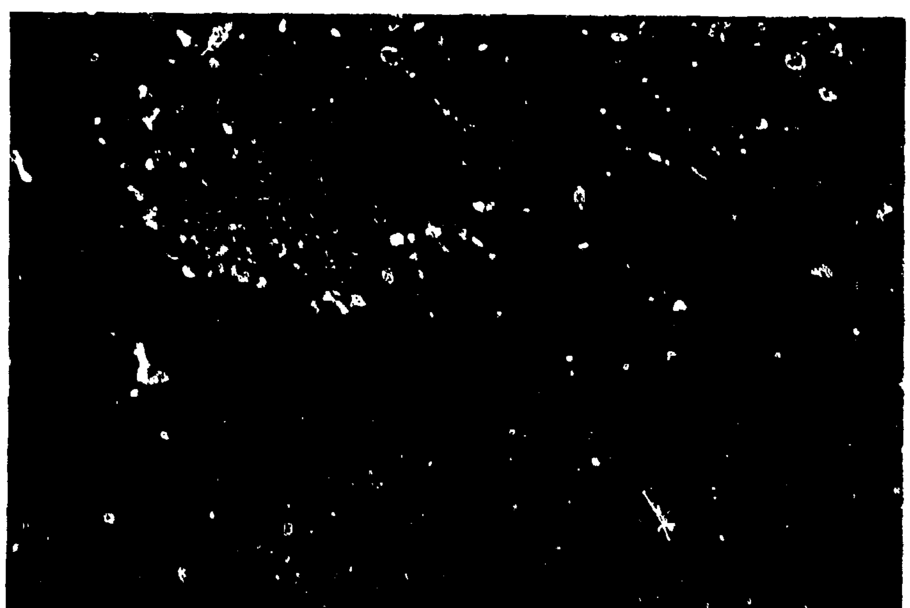

Figure 5 - Nitrocellulose Treated with Dye

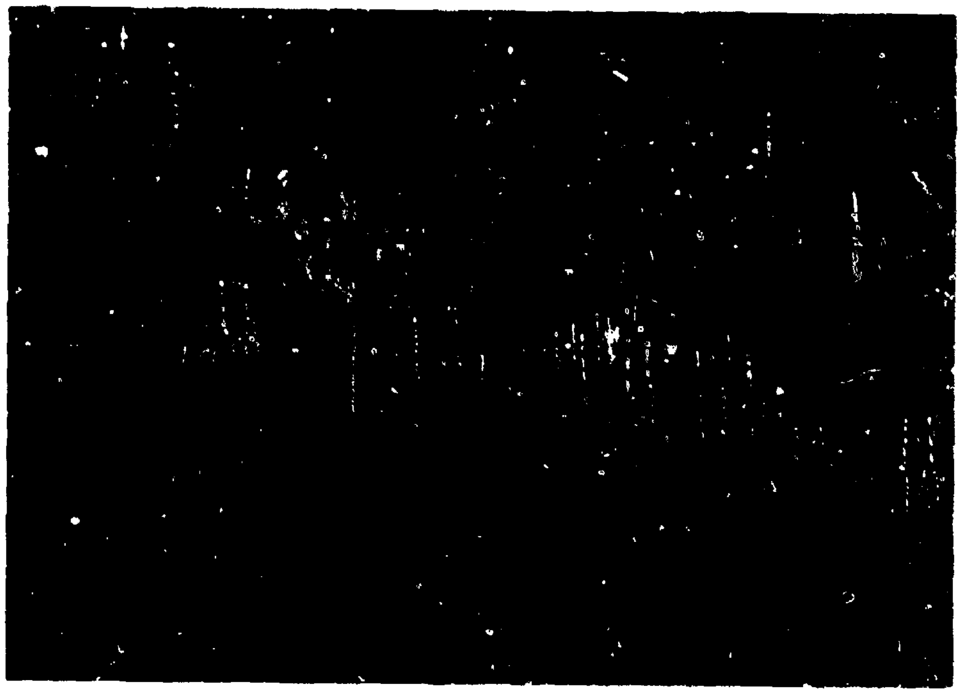

Figure 6 - Cellulose Treated with Dye Solution

Figure 7 - Nitrocellulose and Cellulose Fibers Treated with Dye

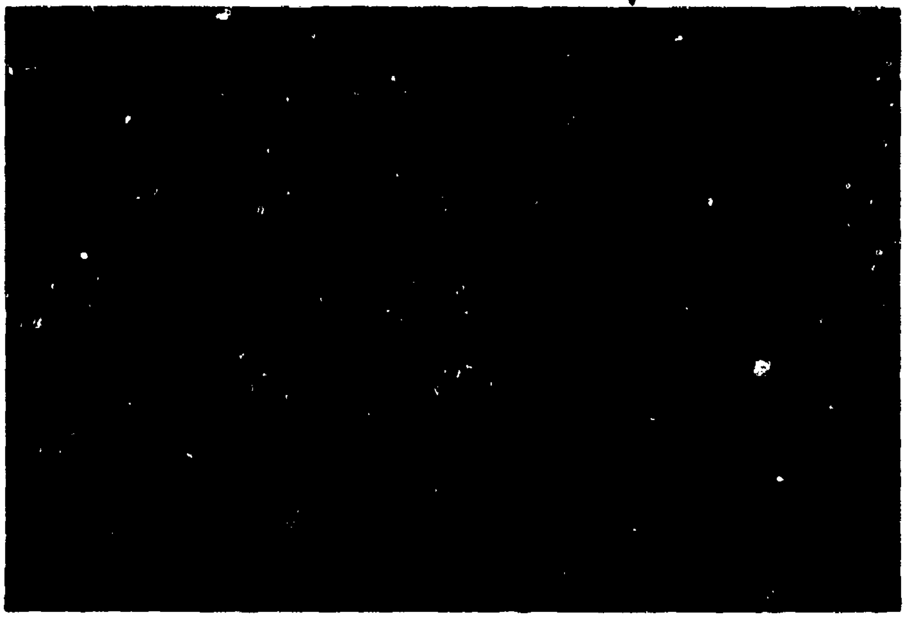

Figure 8 - Cellulose Treated with Stain

Figure 9 - Nitrocellulose Treated with Stain

Figure 10 - Nitrocellulose Treated with Stain

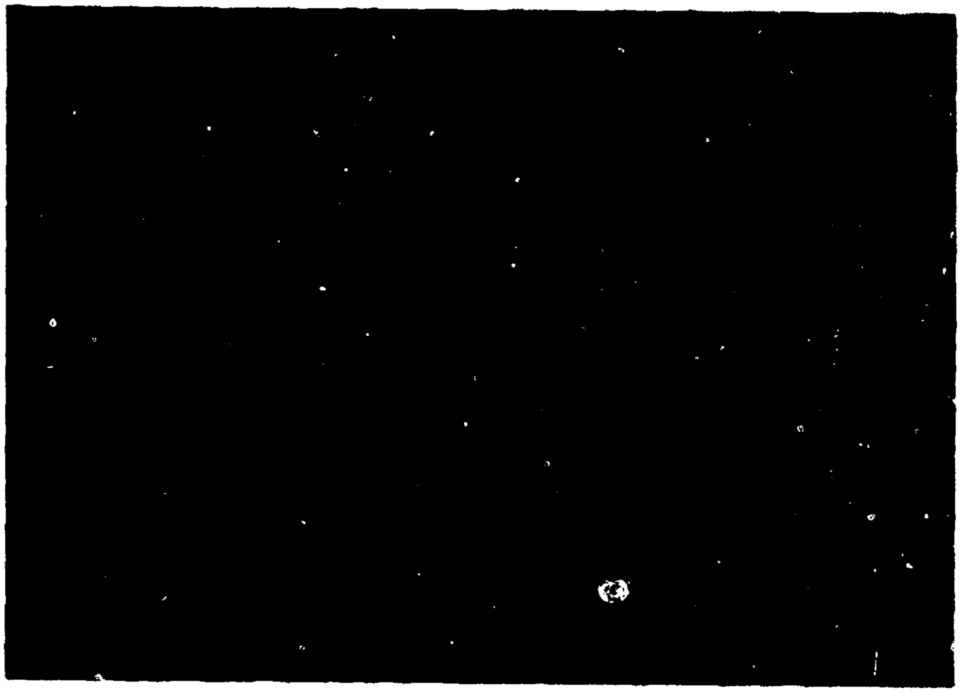

Figure 11 - Mixture of Cellulose and Nitrocellulose with Stain

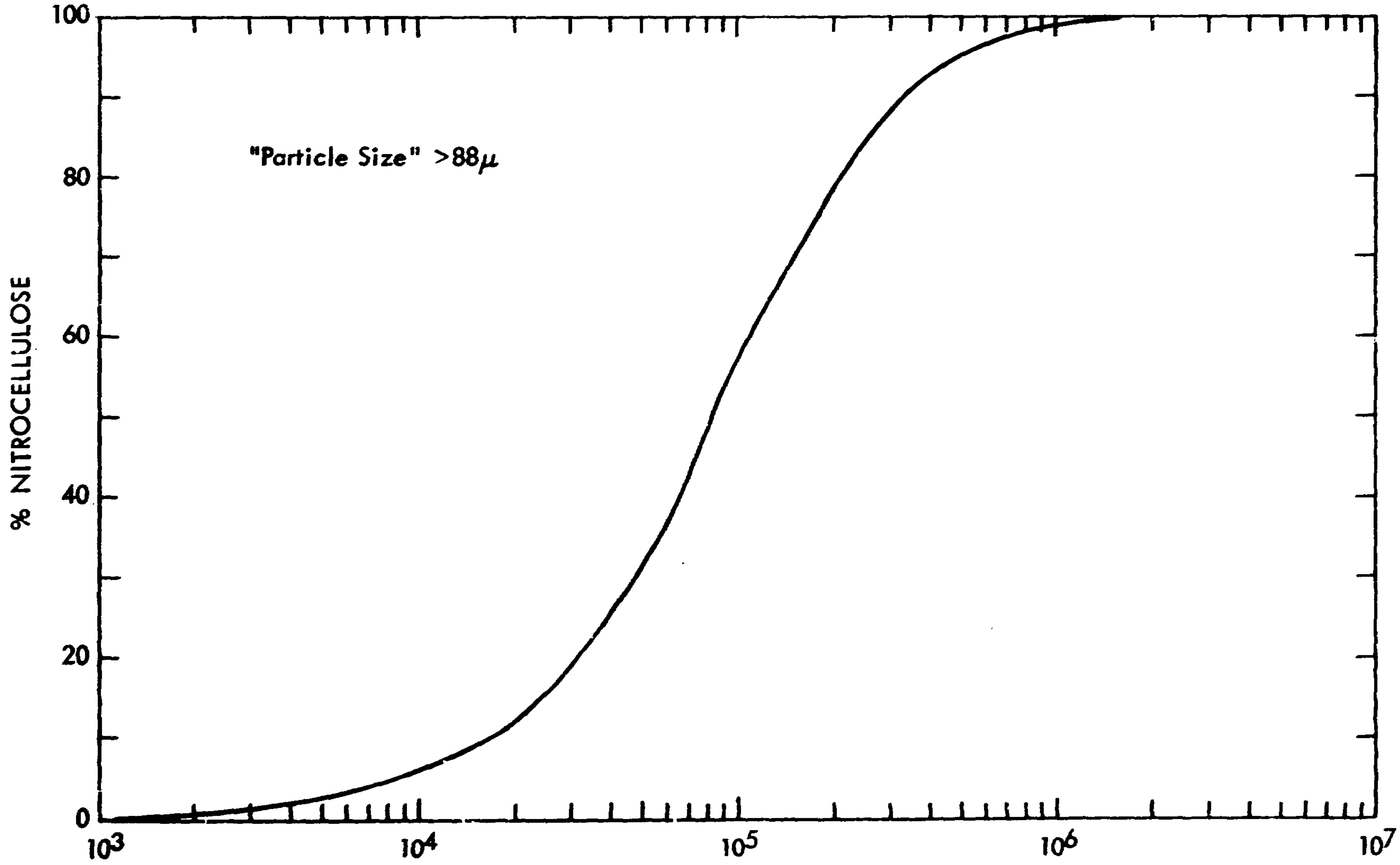

Figure 12 - MOLECULAR WEIGHT

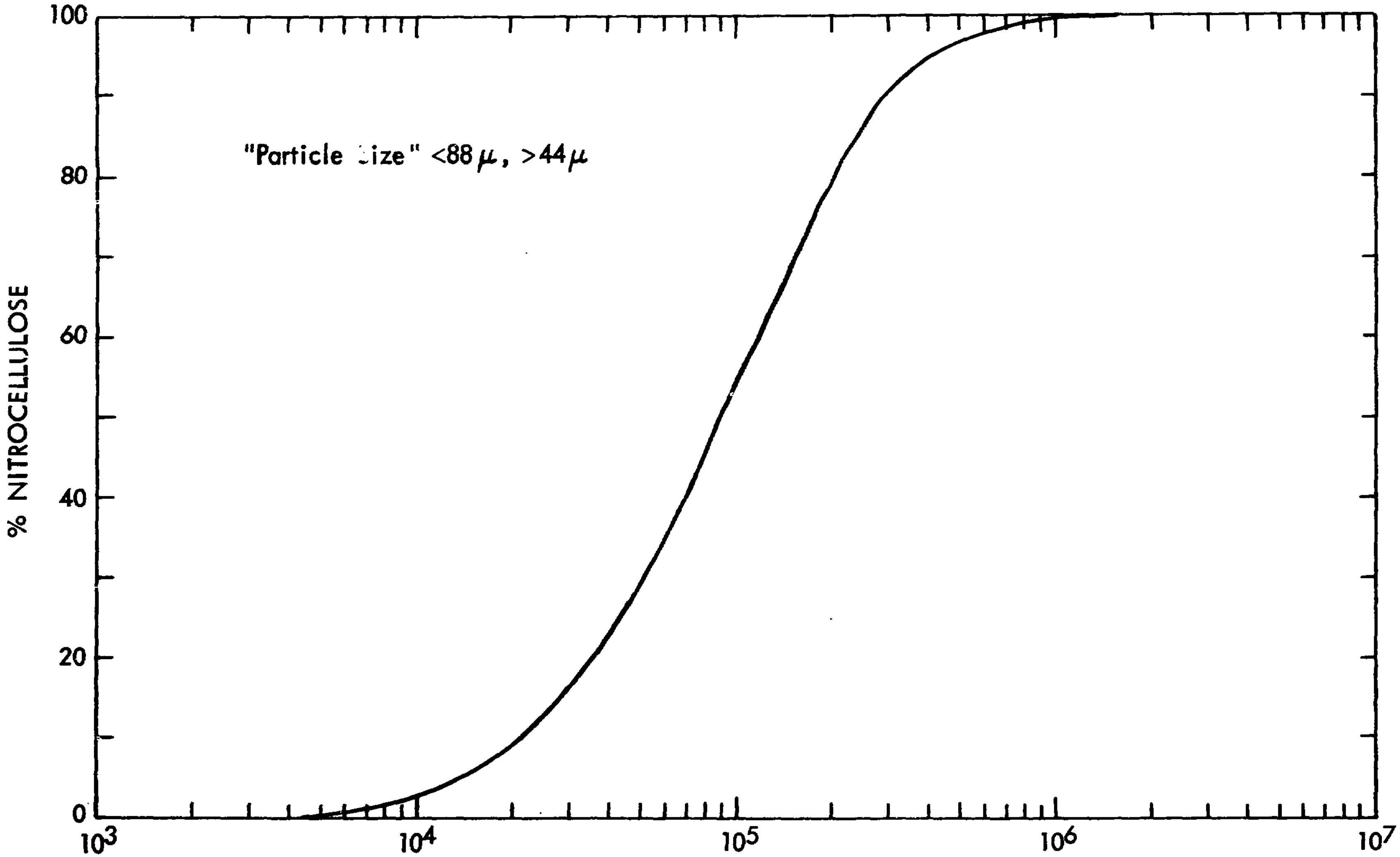

Figure 13 - MOLECULAR WEIGHT

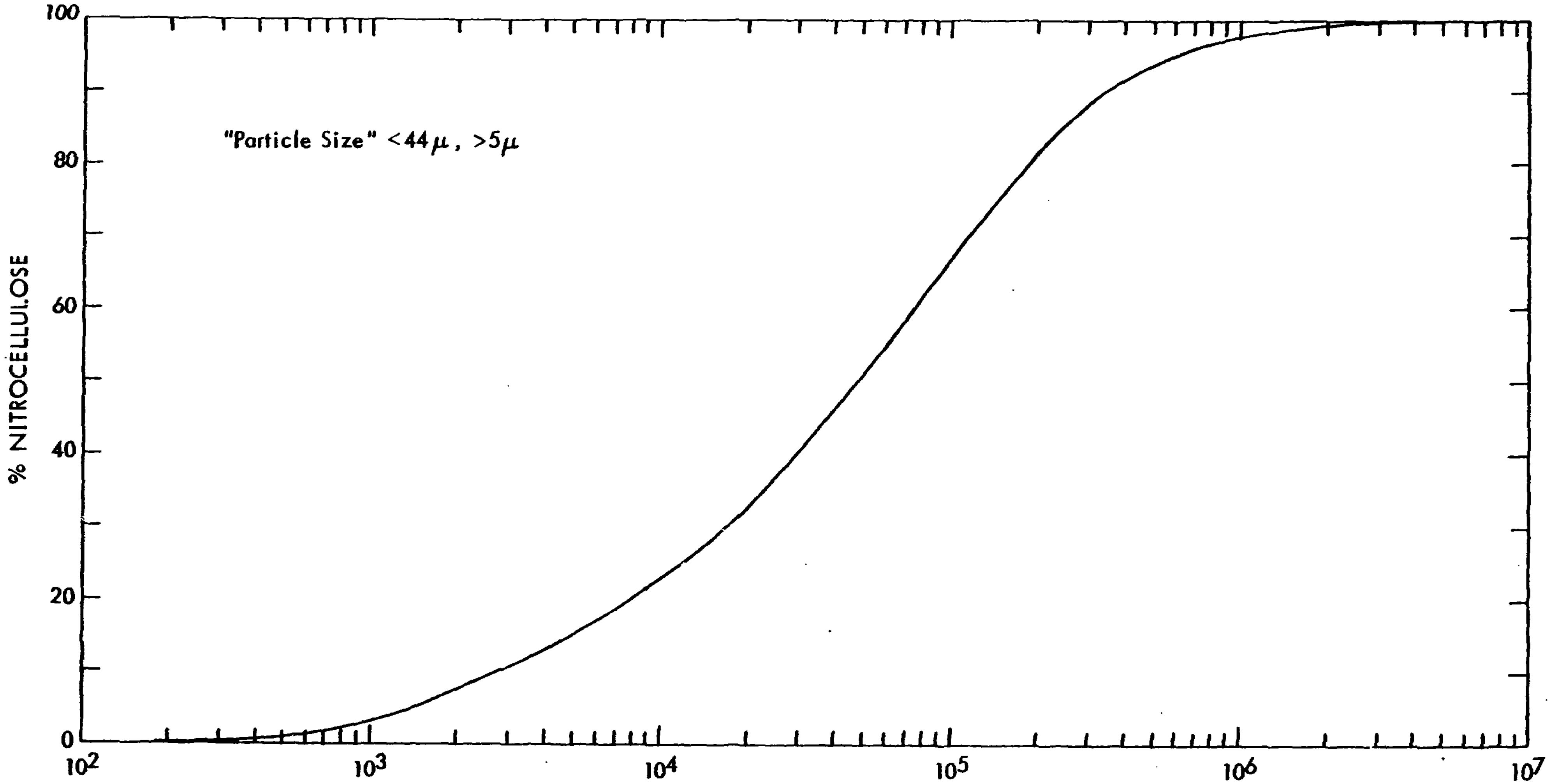

Figure 14 - MOLECULAR WEIGHT

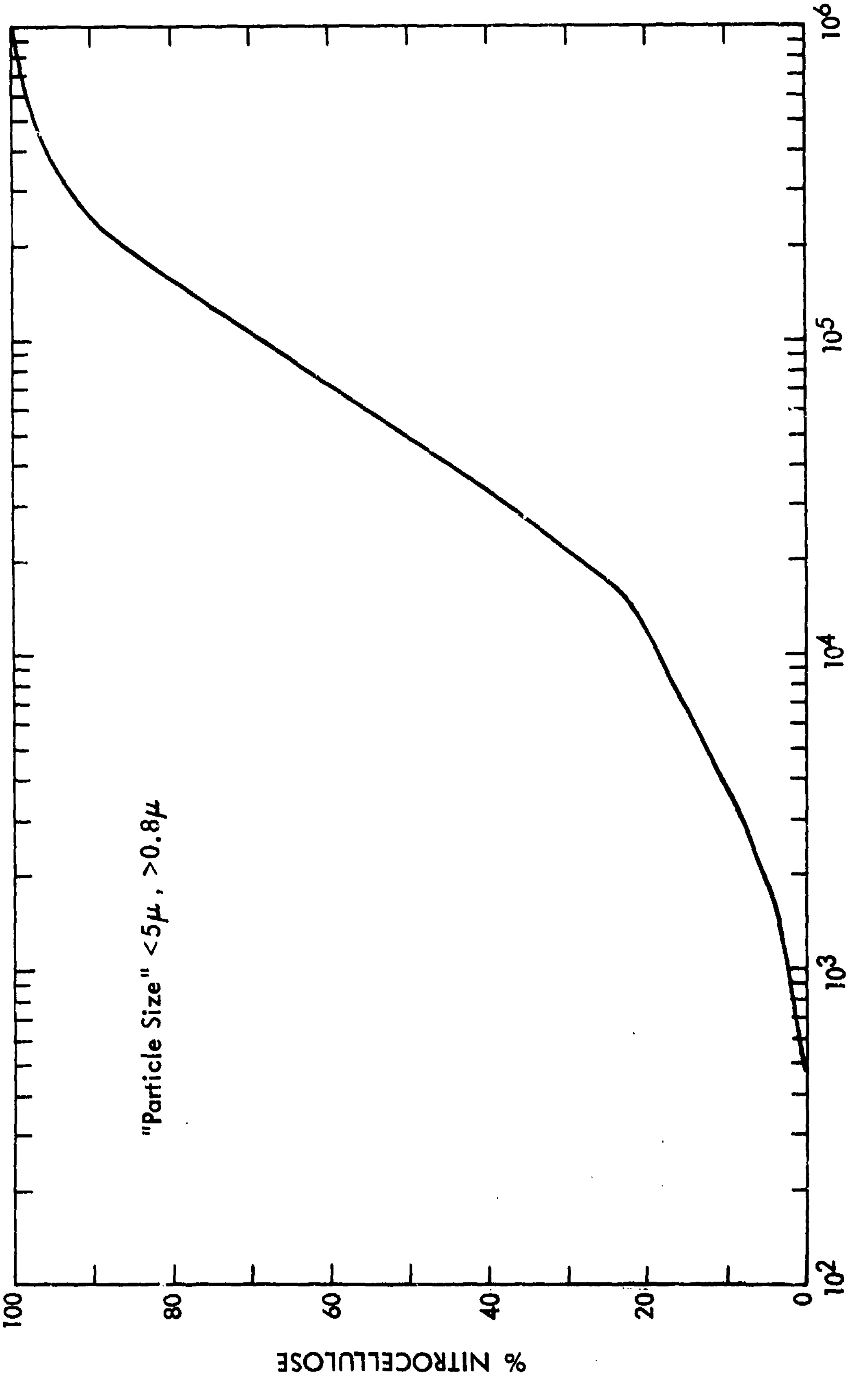

Figure 15 - MOLECULAR WEIGHT